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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT: ROLAND BRUNNER ET AL.-1 (CPA)  
SERIAL NO: 09/425,694 EXAMINER: M. SONG  
FILED: OCTOBER 22, 1999 GROUP: 1765  
TITLE: PROCESS FOR THE WET CHEMICAL TREATMENT OF  
SEMICONDUCTOR WAFERS

COVER LETTER ENCLOSING BRIEF ON APPEAL

MAIL STOP APPEAL BRIEF - PATENTS  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Dear Sir:

Enclosed herewith for filing is the Brief on Appeal in triplicate, along with the required Brief Fee of \$500.00 for a Large Entity. The Commissioner of Patents is hereby authorized to charge any underpayment or credit any overpayment to Deposit Account No. 03-2468.

Respectfully submitted,  
ROLAND BRUNNER ET AL. (CPA)

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Enclosure: Brief on Appeal in triplicate  
Check for \$500.00

I hereby certify that this correspondence is being deposited with the U.S. Postal Service as first class mail in an envelope addressed to: Assistant Commissioner of Patents, P.O. Box 1450, Alexandria, VA 22313-1450, on April 14, 2005.

Ingrid Mittendorf



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**BRIEF ON APPEAL**

MAIL STOP APPEAL BRIEF - PATENTS  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 2313-1450

Dear Sir:

In accordance with the provisions of Rule 192(c), the following items under appropriate headings are provided:

(1) REAL PARTY IN INTEREST:

The real party in interest is the party named in the caption above.

(2) RELATED APPEALS AND INTERFERENCES:

There are no other appeals or interferences known to Appellant, the Appellant's legal representative, or assignee which will directly affect or be directly affected by or have a bearing on the Board's decision in the pending appeal.

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(3) STATUS OF CLAIMS:

Claims 1-9 and 11-15 are being appealed. Claims 1-9 and 11-15 are pending.

(4) STATUS OF AMENDMENTS:

Claims 1-9 and 11 stand rejected under 35 USC 103(a) as being unpatentable over *Pirooz et al.* in view of *Verhaverbeke et al.* and claims 12-15 stand rejected under 35 USC 103 as being unpatentable over *Pirooz et al.* in view of *Verhaverbeke* and further in view of *Berman* or *Davison*. All of the previous amendments have been entered.

(5) SUMMARY OF THE INVENTION:

The present invention relates to a process for the wet chemical treatment of semiconductor wafers, in which the semiconductor wafers are treated with treatment liquids, in particular a process for the cleaning of silicon semiconductor wafers.

It is an object of the present invention to provide a process by which metallic impurities and particles can be removed particularly effectively from semiconductor wafers.

The above object is achieved according to the present invention by providing a process for the wet chemical treatment of semiconductor wafers, in which the semiconductor wafers are

treated with treatment liquids, wherein the semiconductor wafers are firstly treated with an aqueous HF solution, then with an aqueous O<sub>3</sub> solution and finally with an aqueous HCl solution, these treatments forming a treatment sequence.

It has been found that the object is achieved by this treatment sequence, which does not need to be interrupted by rinsing with water or another treatment liquid and is carried out exclusively at a pH which is lower than pH 7. The treatment according to the invention with the treatment liquids indicated is performed in treatment baths. It is preferable to circulate the treatment liquid, that is to take some of this liquid from the corresponding treatment bath and return it back after having been filtered. This saves on outlay for the required chemicals and for deionized water. The addition of fresh water or other liquids to the treatments baths is to be avoided since, when valves are opened, pressure impulses are created and particles can be introduced into the treatment baths. The treatment according to the invention is therefore different from a rinsing treatment, in which fresh treatment liquid is supplied continuously or at intervals.

The present invention is characterized in that the wafers are treated with liquids in a specific sequence of baths, wherein an interruption of the treatment sequence by rinsing with water or another liquid must be strictly avoided.

As to the comparative examples disclosed in the present patent Specification, they clearly show the disadvantageous effect if the sequence is interrupted by a rinsing step with water even if megasonic waves are acting on the water during the rinsing step. Only the claimed combination of treatment steps with the final treatment with an aqueous HCl solution leads to an acceptable low number of particles on the wafers.

#### (6) ISSUES:

A concise statement of the issues presented for review is as follows:

The issues presented in this Appeal is whether, or not, the final rejection of claims 1 to 9 and 11 under 35 U.S.C. 103 as being unpatentable over *Pirooz* in view of *Verhaverbeke*, should be affirmed, or should be reversed, and whether the rejection of claims 12-15 under 35 U.S.C. 102 as being unpatentable over *Pirooz* in view of *Verhaverbeke* and further in view of *Berman* or *Davison* should be affirmed or reversed.

#### (7) GROUPING OF CLAIMS

Claims 1-9 and 11-15 are to be grouped together.

#### (8) ARGUMENT

The above-defined issues are believed to be in error and should be reversed for the following reasons.

On Page 2 of the Office Action, the Patent Examiner has rejected claims 1-9 and 11 under 35 U.S.C. 103(a) as being unpatentable over *Pirooz et al* (EP 0701275) in view of *Verhaverbeke et al* (U.S. Patent No. 6,132,522).

On Page 6 of the Office Action, the Patent Examiner has rejected claims 12-15 under 35 U.S.C. 103(a) as being unpatentable over *Pirooz* (EP 0701275) in view of *Verhaverbeke* (U.S. Patent No. 6,132,522) as applied to claims 1-9 and 11, and further in view of *Berman* (U.S. Patent No. 5,014,737) (newly cited), or *Davison et al* (U.S. Patent No. 5,593,538) (newly cited).

The present invention is directed to a process for the wet chemical treatment of semiconductor wafers with treatment liquids in baths, consisting of the steps of

firstly treating the semiconductor wafers in a bath with an aqueous HF solution containing HF and optionally HCl and optionally a surfactant;

then treating the semiconductor wafers in a bath with an aqueous  $O_3$  solution containing  $O_3$  and optionally HF; and

then treating the semiconductor wafers in a bath with an aqueous HCl solution containing HCl and optionally  $O_3$ ;

whereby these treatment steps form a treatment sequence  $B_2$ , which avoids rinsing with water or another treatment liquid and

the addition of fresh water or other liquids to the treatment baths.

Also, the present invention is directed to a process for the wet chemical treatment of semiconductor wafers with treatment liquids in baths, comprising the steps of

firstly treating the semiconductor wafers in a bath with an aqueous HF solution containing HF and optionally HCl and optionally a surfactant;

then treating the semiconductor wafers in a bath with an aqueous  $O_3$  solution containing  $O_3$  and optionally HF; and

then treating the semiconductor wafers in a bath with an aqueous HCl solution containing HCl and optionally  $O_3$ ;

whereby these treatment steps form a treatment sequence  $B_2$ ; and

circulating the treatment liquids of said baths by taking a part from each of said baths, filtering and returning the part to the corresponding treatment bath.

Furthermore, the present invention is directed to a process for the wet chemical treatment of semiconductor wafers with treatment liquids in baths, comprising the steps of:

firstly treating the semiconductor wafers in a bath with an aqueous HF solution containing HF and optionally HCl and optionally a surfactant;

then treating the semiconductor wafers in a bath with an aqueous  $O_3$  solution containing  $O_3$  and optionally HF; and

then treating the semiconductor wafers in a bath with an aqueous HCl solution containing HCl and optionally  $O_3$ ; and

circulating the treatment liquids of said baths by taking a part from each of said baths, filtering and returning the part to the corresponding treatment bath;

whereby these treatment steps form a treatment sequence  $B_2$ , which avoids rinsing with water or another treatment liquid and the addition of fresh water or other liquids to the treatment baths.

In response to these rejections, it appears that there is still a misunderstanding of the present invention which is based upon a misinterpretation of the teachings of the prior art references. Specifically, the primary reference *Pirooz* only teaches a chemical treatment process in which there are two (2) active, positive steps for metal removal. This begins with the last paragraph in column 2 in *Pirooz* in which an aqueous HF treatment solution is used. Then *Pirooz* includes column 3, lines 8-13, wherein a first water rinsing step is disclosed. This is followed by the disclosure in *Pirooz* column 3, lines 25-35, in which an aqueous ozone treatment step is disclosed. Then there is a second water rinsing step in *Pirooz* column 3, lines 35-40.



The *Verhaverbeke* secondary reference discloses the elimination of water rinse (column 3, lines 15 to 23) between treatments with various chemical reagents for semiconductor cleaning. It is respectfully submitted that there is no explanation given in the Final Office Action that satisfactorily explains why one skilled in the art would modify the *Piroom* primary reference to add a third treatment step with a third active chemical reagent, other than through the use of hindsight based upon the Applicants' own teachings and the Applicants' own disclosure in the present Specification.

On page 7 of the Office Action, the Patent Examiner recognizes that obviousness can only be established by combining or modifying the teachings of the prior art to produce the claimed invention where there is some teaching, suggestion, or motivation to do so found either in the references themselves or in the knowledge generally available to one of ordinary skill in the art. Further, the Patent Examiner points out that regarding the present application, *Verhaverbeke* teaches the elimination of a DI rinse between each chemical treatment step results in significant savings and higher output.

Finally, the Patent Examiner concludes that the higher output and increased saving is sufficient motivation to a person of ordinary skill in the art at the time of the invention to

modify *Pirooz* by eliminating the DI rinse, as taught by *Verhaverbeke*.

If this argumentation is accepted, *Pirooz* would teach a process which comprises firstly treating semiconductor wafers with an aqueous HF solution containing HF; and then treating the semiconductor wafers in a bath with an aqueous O<sub>3</sub> solution containing O<sub>3</sub>.

*Pirooz* would not teach treating the semiconductor wafers in a bath with an aqueous HCl solution containing HCl. This is also accepted by the Patent Examiner on Page 3 of the Office Action. However, at this stage, the argumentation of the Patent Examiner becomes more non-convincing. The Patent Examiner argues that *Verhaverbeke* teaches that a rinse fluid may be DI water or a very dilute aqueous solution of hydrochloric acid to prevent metallic deposition on the surface (see page 8 of the Office Action). The Patent Examiner then concludes that this teaching would be sufficient motivation to modify *Pirooz* by using a solution of hydrochloric acid as a rinse in place of the DI rinse to prevent metallic deposition.

This is an apparently wrong conclusion, since *Verhaverbeke* teaches a DI rinse and a rinse with a very dilute aqueous solution of hydrochloric acid as equivalent steps (col. 5, lines 5-9). *Verhaverbeke* does not contain any teaching or motivation

for preferring a rinse with a very dilute aqueous solution of hydrochloric acid to a rinse with DI. Therefore, if the above argument is accepted that *Verhaverbeke* suggests eliminating a DI rinse, the same argument must hold for the rinse with a very dilute aqueous solution of hydrochloric acid. Thus, *Verhaverbeke* clearly does not suggest modifying *Pirooz* by using a solution of hydrochloric acid, because this step would act in opposition to a higher output and an increased savings.

In addition to all of the above, it is respectfully submitted that any attempt to combine the teachings of the *Pirooz* primary reference and the *Verhaverbeke* secondary reference would destroy the teachings of the *Pirooz* primary reference. This is impermissible under 35 U.S.C. 103.

*Pirooz* in column 1, in lines 55 to 59 and in column 2, in lines 1 to 2, discloses that the process of *Pirooz* comprises the steps of contacting the surface of the silicon wafer with an aqueous solution containing hydrofluoric acid to remove metals from the wafer surface, and contacting the hydrofluoric acid treated wafers with ozonated water to grow a hydrophilic oxide layer on the surface of the silicon wafer.

*Pirooz* in column 3, in lines 14 to 30, discloses that the rinsed wafers are contacted with high purity ozonated water, i.e., deionized water containing ozone and having a concentration

of no more than about 0.01 parts per billion (weight/volume) of each of iron, chromium, titanium and other metals which will degrade minority carrier lifetime, to produce a wafer having a hydrophilic oxide surface. The resulting silicon oxide layer preferably has a thickness of about 0.6 to about 1.5 nanometers and a concentration of no more than  $1 \times 10^9$ , more preferably no more than about  $1 \times 10^8$  atoms/cm<sup>2</sup> of each of iron, chromium, titanium and other metals which degrade minority carrier lifetime. Silicon oxide layers of this thickness and purity may be produced by immersing the wafers in a bath containing about 0.1 parts per million (ppm) to 50 ppm of ozone, preferably about 10 to about 15 ppm ozone, at a temperature of about 0°C to about 60°C, preferably room temperature, for a period of at least about 0.1 minutes, preferably about 2 to about 10 minutes.

Therefore, any further treatment of the hydrophilic oxide layer on the surface of the silicon wafer of *Pirooz* as taught by *Verhaverbeke* would destroy the properties of the surface, which surface is required by *Pirooz*. Such a radical reconstruction of the prior art references that will destroy the hydrophilic oxide layer surface as mandated by *Pirooz* is impermissible according to 35 U.S.C. 103. This destruction of the teachings of the primary reference to *Pirooz* prevents the Patent Examiner from establishing a prima facie case of obviousness under 35 U.S.C. 103.

Therefore, the PTO must provide an explanation as to why a person skilled in the art would have modified *Pirooz*, based upon *Verhaverbeke* to produce the claimed three step process devoid of any water rinsing steps.

The comparative testing on pages 4 and 5 of the Specification shows a significant improvement in reducing LPD (lightpoint defects).

The comparison below of an Invention example with comparative examples shows the advantageous effect of the present invention.

The Table contains the results (3 sigma values) of conventional counts of particles of specific size (LPD=light point defects), which were in each case carried out on 20 polished semiconductor wafers after a wet chemical treatment sequence and drying which was the same for all the semiconductor wafers. The attached Table appears on page 5 of the present Specification.

The example (B) of the Invention comprised the treatment sequence: HF bath, deionized water/ozone bath, HCl bath with megasonic exposure.

Comparative Example 1 (C1) comprised the treatment sequence: HF bath, deionized water/ozone bath, rinsing with deionized water outside the bath. This corresponds to *Pirooz*, set "B" without megasonics.

Comparative Example 2 (C2) comprised the treatment sequence: HF bath, deionized water/ozone bath with subsequent rinsing using deionized water in the bath and megasonic exposure. This corresponds to *Pirooz* set "A" with megasonics.

*Berman (U.S. Patent No. 5,014,737)* in column 2, lines 50 to 68, discloses a recirculating chemical bath arrangement which is designed to overcome the deficiencies with regard to the two basic forms of quartz tank recirculating systems. This includes a quartz process tank having a trough extending about the top portion of the outer walls of the tank. The system also includes a pump/filter circuit which pumps the chemical solution into the bottom of the process tank, the liquid flowing over the tank walls into the trough. Also the process tank is provided with heater units secured to outer surface thereof. However, rather than connecting the pump/filter input directly to the trough, there is a second, smaller chamber secured to the end of the process tank and disposed to receive liquid flow from the trough as well as from the process tank. The second chamber includes a fitting at the bottom thereof to supply liquid to the pump/filter circuit.

This process and apparatus of *Pirooz* in view of *Berman* will produce massive contamination of the treatment baths, leading to the high increase in LPD's for C1 and C2, as shown in the attached Table. For example, according to the Invention, the LPD's are 7, or 30, depending upon the size of the LPD. For *Pirooz* in view of *Berman*, the corresponding number of LPD's would be 13 for C1 and would be 50 for C2, if B of the Invention were 7. Again, for *Pirooz* in view of *Berman*, the corresponding number of LPD's would be 50 for C1 and would be 140 for C2, if B were 30. Thus, one skilled in the art would never combine the teachings of *Berman* with either *Pirooz* alone, or *Pirooz* in combination with *Verhaverbeke*. To do so would produce the extremely undesirable results discussed above, in which the greatly increased number of LPD's produced is completely undesirable.

*Davison* (U.S. Patent No. 5,593,538) in column 1, lines 54 to 67 and column 2, in lines 1 to 5, discloses a method for etching a substrate. More particularly, *Davison* provides a wet etching process for an oxide layer on a semiconductor material.

In a wet etching process in accordance with *Davison*, polycrystalline silicon is etched when it is exposed to an etchant. The etching of the polycrystalline silicon may cause open polycrystalline silicon lines in some areas as well as reliability problems by creating very thin polycrystalline

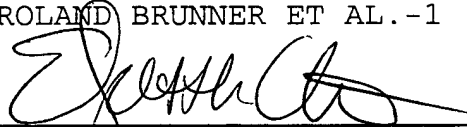
silicon lines. An objective of *Davison* is to provide an etching process that does not etch a conductive structure such as, for example, a polycrystalline silicon structure, on a semiconductor wafer. Through an extensive investigation, it has been discovered that the etching of the polycrystalline silicon structure is caused by oxygen dissolved in the etchant. Therefore, *Davison* provides a method for protecting the conductive structure on a substrate during a wet etching process by purging away oxygen dissolved in the etchant.

This process and apparatus of *Pirooz* in view of *Davison* will produce massive contamination of the treatment baths leading to the huge increase in LPD's for C1 and C2, as shown in the attached Table. For example, according to the Invention, the LPD's are 7, or 30, depending upon the size of the LPD. For *Pirooz* in view of *Davison*, the corresponding number of LPD's would be 13 for C1 and would be 50 for C2, if B of the Invention were 7. Again, for *Pirooz* in view of *Davison*, the corresponding number of LPD's would be 50 for C1 and would be 140 for C2, if B were 30. Thus, one skilled in the art would never combine the teachings of *Davison* with either *Pirooz* alone, or *Pirooz* in combination with *Verhaverbeke*. To do so would produce the extremely undesirable results discussed above, in which the greatly increased number of LPD's produced is completely undesirable.



Accordingly, Applicant submits that claims 1-9 and 11-15 are patentable over the cited references, taken either singly or in combination. Early allowance of the claims is respectfully requested.

Respectfully submitted,  
ROLAND BRUNNER ET AL.-1 (CPA)



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Enclosure: Appendix containing appealed claims 1-9 and 11-15  
Copy of Table

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Ingrid Mittendorf

(9) APPENDIX

The Appealed claims are as follows:

Claim 1.

A process for the wet chemical treatment of semiconductor wafers with treatment liquids in baths, consisting of the steps of

firstly treating the semiconductor wafers in a bath with an aqueous HF solution containing HF and optionally HCl and optionally a surfactant;

then treating the semiconductor wafers in a bath with an aqueous  $O_3$  solution containing  $O_3$  and optionally HF; and

then treating the semiconductor wafers in a bath with an aqueous HCl solution containing HCl and optionally  $O_3$ ;

whereby these treatment steps form a treatment sequence  $B_2$ , which avoids rinsing with water or another treatment liquid and the addition of fresh water or other liquids to the treatment baths.

Claim 2.

The process as claimed in claim 1,

wherein the treatment sequence  $B_2$  is preceded by a treatment  $B_1$  of the semiconductor wafers with an aqueous SC-1 solution.

Claim 3.

The process as claimed in claim 1,

wherein the treatment sequence  $B_2$  is followed by a treatment  $B_3$  comprising drying the semiconductor wafers.

Claim 4.

The process as claimed in claim 3,

wherein the treatment of the semiconductor wafers is sequenced according to the term  $m \cdot (B_1 + B_2) + B_3$ ,

$m$  being an integer number and the treatment  $B_1$  and the treatment sequence  $B_2$  being carried out in succession, and

this taking place  $m$  times, before the drying treatment  $B_3$  is performed.

Claim 5.

The process as claimed claim 1,

wherein in treatment sequence  $B_2$ , the aqueous HF solution contains HF in a concentration of from 0.001% to 2% by weight and optionally HCl in a concentration of up to 2% by weight and optionally a surfactant; and

wherein all percents by weight are based upon the total solution weight.

Claim 6.

The process as claimed in claim 1,

wherein in treatment sequence B<sub>2</sub>, the aqueous O<sub>3</sub> solution contains O<sub>3</sub> in a concentration of from 1 ppm to 30 ppm and is optionally exposed to megasonic waves.

Claim 7.

The process as claimed in claim 1,

wherein the treatment liquid used last in the treatment sequence B<sub>2</sub> contains ozone and is optionally exposed to megasonic waves.

Claim 8.

The process as claimed in claim 3,

wherein the drying treatment is carried out using a step selected from the group consisting of centrifuging, using hot water, using isopropanol, and using marangoni principle.

Claim 9.

The process as claimed in claim 2,

wherein in treatment B<sub>1</sub> the aqueous SC-1 solution contains a liquid selected from the group consisting of NH<sub>4</sub>OH and H<sub>2</sub>O<sub>2</sub>, and TMAH (= tetramethylammonium hydroxide) and H<sub>2</sub>O<sub>2</sub>.

Claim 10 (Cancelled).

Claim 11.

A process for the wet chemical treatment of semiconductor wafers with treatment liquids in baths, consisting of the steps of

firstly treating the semiconductor wafers in a bath with an aqueous HF solution containing HF and optionally HCl and optionally a surfactant;

then treating the semiconductor wafers in a bath with an aqueous O<sub>3</sub> solution containing O<sub>3</sub> and optionally HF; and

then treating the semiconductor wafers in a bath with an aqueous HCl solution containing HCl and optionally O<sub>3</sub> with exposure to megasonic waves,

whereby these treatment steps form a treatment sequence B<sub>2</sub>, which avoids rinsing with water or another treatment liquid and the addition of fresh water or other liquids to the treatment baths.

Claim 12.

A process for the wet chemical treatment of semiconductor wafers with treatment liquids in baths, comprising the steps of

firstly treating the semiconductor wafers in a bath with an aqueous HF solution containing HF and optionally HCl and optionally a surfactant;

then treating the semiconductor wafers in a bath with an aqueous O<sub>3</sub> solution containing O<sub>3</sub> and optionally HF; and

then treating the semiconductor wafers in a bath with an aqueous HCl solution containing HCl and optionally O<sub>3</sub>;

whereby these treatment steps form a treatment sequence B<sub>2</sub>;  
and

circulating the treatment liquids of said baths by taking a part from each of said baths, filtering and returning the part to the corresponding treatment bath.

Claim 13.

A process for the wet chemical treatment of semiconductor wafers with treatment liquids in baths, comprising the steps of

firstly treating the semiconductor wafers in a bath with an aqueous HF solution containing HF and optionally HCl and optionally a surfactant;

then treating the semiconductor wafers in a bath with an aqueous O<sub>3</sub> solution containing O<sub>3</sub> and optionally HF; and

then treating the semiconductor wafers in a bath with an aqueous HCl solution containing HCl and optionally O<sub>3</sub> with exposure to megasonic waves;

whereby these treatment steps form a treatment sequence B<sub>2</sub>;  
and

circulating the treatment liquids of said baths by taking a part from each of said baths, filtering and returning the part to the corresponding treatment bath.

Claim 14.

A process for the wet chemical treatment of semiconductor wafers with treatment liquids in baths, comprising the steps of  
firstly treating the semiconductor wafers in a bath with an aqueous HF solution containing HF and optionally HCl and optionally a surfactant;

then treating the semiconductor wafers in a bath with an aqueous  $O_3$  solution containing  $O_3$  and optionally HF; and

then treating the semiconductor wafers in a bath with an aqueous HCl solution containing HCl and optionally  $O_3$ ; and

circulating the treatment liquids of said baths by taking a part from each of said baths, filtering and returning the part to the corresponding treatment bath;

whereby these treatment steps form a treatment sequence  $B_2$ , which avoids rinsing with water or another treatment liquid and the addition of fresh water or other liquids to the treatment baths.

Claim 15.

A process for the wet chemical treatment of semiconductor wafers with treatment liquids in baths, comprising the steps of  
firstly treating the semiconductor wafers in a bath with an aqueous HF solution containing HF and optionally HCl and optionally a surfactant;

then treating the semiconductor wafers in a bath with an aqueous  $O_3$  solution containing  $O_3$  and optionally HF; and

then treating the semiconductor wafers in a bath with an aqueous HCl solution containing HCl and optionally O<sub>3</sub> with exposure to megasonic waves; and

circulating the treatment liquids of said baths by taking a part from each of said baths, filtering and returning the part to the corresponding treatment bath;

whereby these treatment steps form a treatment sequence B<sub>2</sub>, which avoids rinsing with water or another treatment liquid and the addition of fresh water or other liquids to the treatment baths.





TABLE  
BRUNNER ET AL -I-CPA SHEET 1 OF 1  
SERIAL NO.: 09/425,694  
FILED: OCTOBER 22, 1999 GROUP: 1765  
EXAMINER: M. SONG  
TITLE: PROCESS FOR THE WET TREATMENT  
OF SEMICONDUCTOR WAFERS



LPD ( $\mu\text{M}$ )	>0.3	>0.2	>0.16	>0.12
	Number	Number	Number	Number
B	3	7	30	480
C1	4	13	50	550
C2	10	50	140	550